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A METHOD TO OBTAIN DIFFUSE REFLECTANCE MEASUREMENTS FROM 1.0 TO 3.0 Micrometers USING A CARY 171 SPECTROPHOTOMETER.

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This report describes a way to perform diffuse reflectance measurements over the 1.0 to 3.0μm spectral interval by using a Cary 17I Spectrophotometer equipped with inexpensive standard accessory cell-space integrating spheres. The method involves coating the cell-space spheres with a powder of pure grade quenched sulfur with carbon disulfide used as a solvent and making a minor alteration in an electronic amplifier. A spectrum of ground gypsum crystal in the 2.6 to 3.0μm spectral region and a spectrum of a layer of Hectorite clay in the 1.0 to → next page

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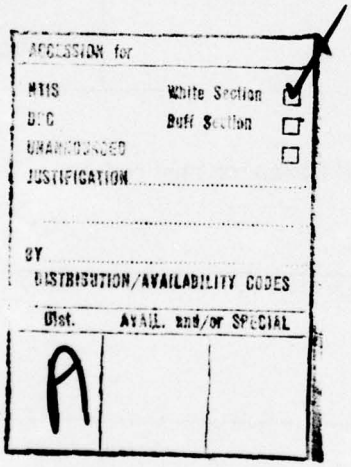
3.0 μm spectral region are presented to illustrate the applicability of the above technique for making diffuse reflectance measurements in the near infrared.



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INTRODUCTION

Optical constants of the atmospheric particulate matter are needed because of the Army's interest in the propagation of electromagnetic radiation in the visible and near infrared portion of the spectrum. Since transmission measurements do not yield acceptable techniques for determining the absorption coefficient of particulate matter suspended in the atmosphere, diffuse reflectance techniques have been developed and applied to the problem [1,2]. These techniques have been used in the 0.4 to 2.5μm spectral region for quantitative determinations of the absorption coefficients of clay minerals [3,4] and have also been used in the 0.3 to 1.1μm spectral region on atmospheric dust [5]. The absorption coefficients for the very strong water band absorption region from 2.7 to 3.0μm have not been determined for powdered material. This absorption region is present in the clay minerals which make up a significant portion by mass of the atmospheric dust.

The limiting factor in the useful spectral range of diffuse reflectance measurements has been the reflectance of the white standard (the sphere coating material and/or the reference material) which determines both the efficiency of the integrating sphere and the applicability of the white standard as a diluting mix for the quantitative measurements of strongly absorbing powders. Recently, a highly refined barium sulfate powder has been used in diffuse reflectance measurements. However, barium sulfate slightly absorbs water, and therefore, its reflectance is poor beyond about 2.0μm in the near infrared. As a diluent its absorption now becomes nontrivial and its effective spectral range of use is reduced to about 1.7μm [6]. As a sphere coating it is usable out to about 1.5μm for the small, inexpensive commercially available Cary accessory cell-space integrating spheres [7] and can be used to about 2.5μm in larger more efficient spheres [3,4]. Skelensky et al. [8] have extended the usable spectral range of the Cary accessory integrating spheres to about 2.5μm by coating the spheres with an alumina powder instead of barium sulfate. They used a 0.01% solution of carboxylmethylcellulose (CMC) as a binder. However, the CMC binder contains water and therefore reduces the use of the coating - especially in the 2.5 to 3.0μm spectral range.

The purpose of this report is to describe a way of extending the spectral range of applicability of the inexpensive Cary accessory cell-space integrating spheres to about 3.0μm by using a Cary 17I spectrophotometer with slight modification to an electronic amplifier and by using a more highly reflecting, anhydrous white standard as a sphere coating.

PROCEDURE

Initially, a sphere coating of aluminum oxide particles with 0.01% CMC binder as in the work of Skelensky et al. was tried, but there was

insignificant signal beyond 2.5μm to operate the slit servomechanism. As an alternative to the aluminum oxide and CMC coating, powders of flowers of sulfur and a pure grade quenched sulfur which have been shown to have a high reflectance in the infrared [9] were tried. The sulfur has a lower spectral limit of 0.5μm where it begins to absorb strongly. The cell-space spheres were first cleaned and polished. Then flowers of sulfur were mixed with carbon disulfide and sprayed onto the spheres with a paint sprayer. The flowers of sulfur are about 30% μ-form sulfur, which is insoluble in carbon disulfide. A part of the sulfur went into solution and recrystallized to form a binder after it was sprayed on the sphere surfaces. Several coatings were applied at about 15-minute intervals to allow the carbon disulfide to evaporate. The final layer on the surface was very sturdy and about 3 mm thick. This coating on the spheres allowed sufficient signal to operate the slit servomechanism to 2.72μm.

A sample of pure grade quenched sulfur (obtained from the Stauffer Chemical Company and referred to as Crystex) was tried in an effort to improve the above results. According to the manufacturer, the Crystex is a polymer-type sulfur with a molecular weight of about 100,000 and is about 87-93% insoluble in carbon disulfide. Its particle size is about 3μm and its index of refraction is about 1.9. Because Crystex is less soluble in CS_2 than the flowers of sulfur, it sprayed into a finer, more powdery layer - somewhat less sturdy than the flowers of sulfur; but it was adequately bound into a layer which was about 2-3 mm thick. When the spheres were coated with this substance the Cary 17I slit servomechanism operated out to a wavelength of 2.80μm and there was pen response at 3.0μm. At the other end of the spectrum the spheres provided satisfactory operation down to 0.5μm. However, for this particular model of the Cary 17I, the grating was blazed at 1.6μm. Therefore, in the infrared region from 0.7 to 1.0μm the slit servo required a higher slit control setting and had a net result of increasing the noise level, widening the spectral bandwidth, and increasing the scan time.

To check the coating procedure several glass plates were spray coated to a thickness of about 3 mm with the flowers of sulfur and with the Crystex. First, a slightly yellowed (decomposed) CS_2 was used as the solvent. The reflectance of these first layers was measured compared to a reference sample dish full of flowers of sulfur. In the 2.1 to 2.8μm spectral region these layers had a reflectance of about 60% relative to the reference. Next, pure CS_2 was used and a reflectance of about 98-99% was obtained, thus indicating that care must be used in handling the CS_2. It was observed during the coating procedure that the texture of the coating could be varied from fairly even to very lumpy depending on the spraying technique. This effect was tested by spraying several layers of Crystex with the texture varying from smooth to very lumpy. The smooth layers produced a reflectance of 98 to 99% while the lumpy layers produced a shadow effect which reduced the reflectance to about 85%.

In putting the final coating on the integrating spheres, the above observations were used as guidelines. Crystex was chosen as the sulfur to use because it sprayed into a smooth, even, powdery surface and was less affected by the CS_2. Another consideration in its favor is its cohesiveness - it can be pressed into a sample dish and used in an upside down position. This is the configuration of the sample for the cell-space spheres. A pure grade CS_2 was used and a smooth layer of Crystex was sprayed to a thickness of about 3 mm onto each of the two cell-space integrating spheres.

To further extend the range of the technique, a minor electronic change was made in the sample/reference amplifier for the slit servo [10]. During this phase of the work the photometric signal was monitored with an oscilloscope. The gain of the amplifier was increased by a factor of 4 or 5. This caused a corresponding increase in the noise level which was somewhat compensated for by a very slow scan speed and higher pen period. Slit response was obtained to 3μm wavelength. The increased noise level is only necessary from 2.8 to 3.0μm since the slit gain can be reduced at wavelengths of 2.8μm and less.

RESULTS AND CONCLUSIONS

A diffuse reflectance spectrum using the equipment described above is shown in Figure 1 for the 2.6 to 3.0μm spectral interval. The sample is a gypsum crystal coarsely ground with a mortar and pestle. Gypsum was selected because it has strong absorption lines at 2.816μm and 2.938μm. The ground gypsum was diluted with Crystex in a dilution of about one part gypsum to 100 parts Crystex. This dilution is necessary because the absorption in this region is so strong that the reflectance spectrum is effectively zero and no structure can be seen. Since the Crystex has no absorption in this region and has a very high reflectance (on the order of 99%), it can be mixed with the sample to act as a matrix. That the absorption lines can be clearly seen demonstrates the applicability of the technique out to 3.0μm in wavelength. The noise level in the figure is about 5 to 6% in the 2.8 to 3.0μm region and about 2 to 3% in the 2.6 to 2.8μm region. In the 3.0 to 2.8μm region the spectrum was run with a scan speed of 0.05 nm/sec, a pen period of 25, and with a spectral bandwidth of about 3.5 to 4.0 nm. From 2.0 to 2.6μm the scan speed was 0.2 nm/sec and from 2.5μm on down, it was 0.5 nm/sec. A potassium bromide pellet transmission spectrum of this same sample is shown in Figure 2 for comparison. Figure 3 shows a smoothed spectrum of a layer of Hectorite clay in the spectral region 1.0 to 2.5μm. The noise level is about 1% in the 1.0 to 2.6μm region. Note the readily recognizable absorption bands in the 1.4, 1.9, 2.3, and 2.7μm wavelength regions due to the O-H bonds in the structure of the Hectorite. Since this sample was not diluted, detail in the 2.6 to 3.0μm region cannot be seen easily. Note that the gypsum diffuse reflectance spectrum is

consistent with normal KBr pellet transmission spectrum in the spectral region, indicating that the instrument can satisfactorily measure diffuse reflectance spectra as desired, using the inexpensive cell-space accessory integrating spheres, modified as described in this report.

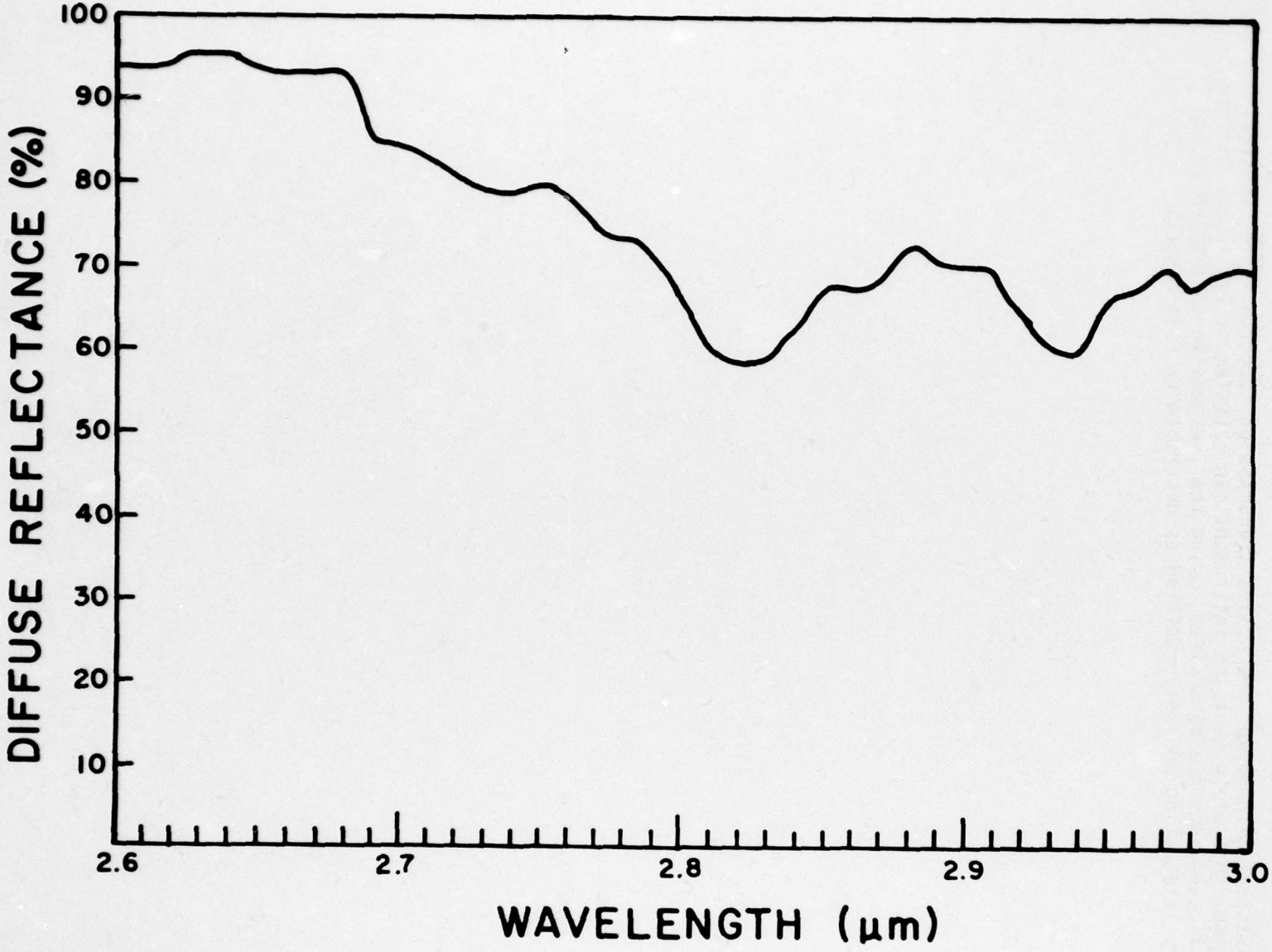

FIGURE 1. DIFFUSE REFLECTANCE SPECTRUM OF GROUND GYPSUM CRYSTAL DILUTED WITH CRYSTEX SULFUR IN THE 2.6 TO 3.0 μm SPECTRAL REGION.

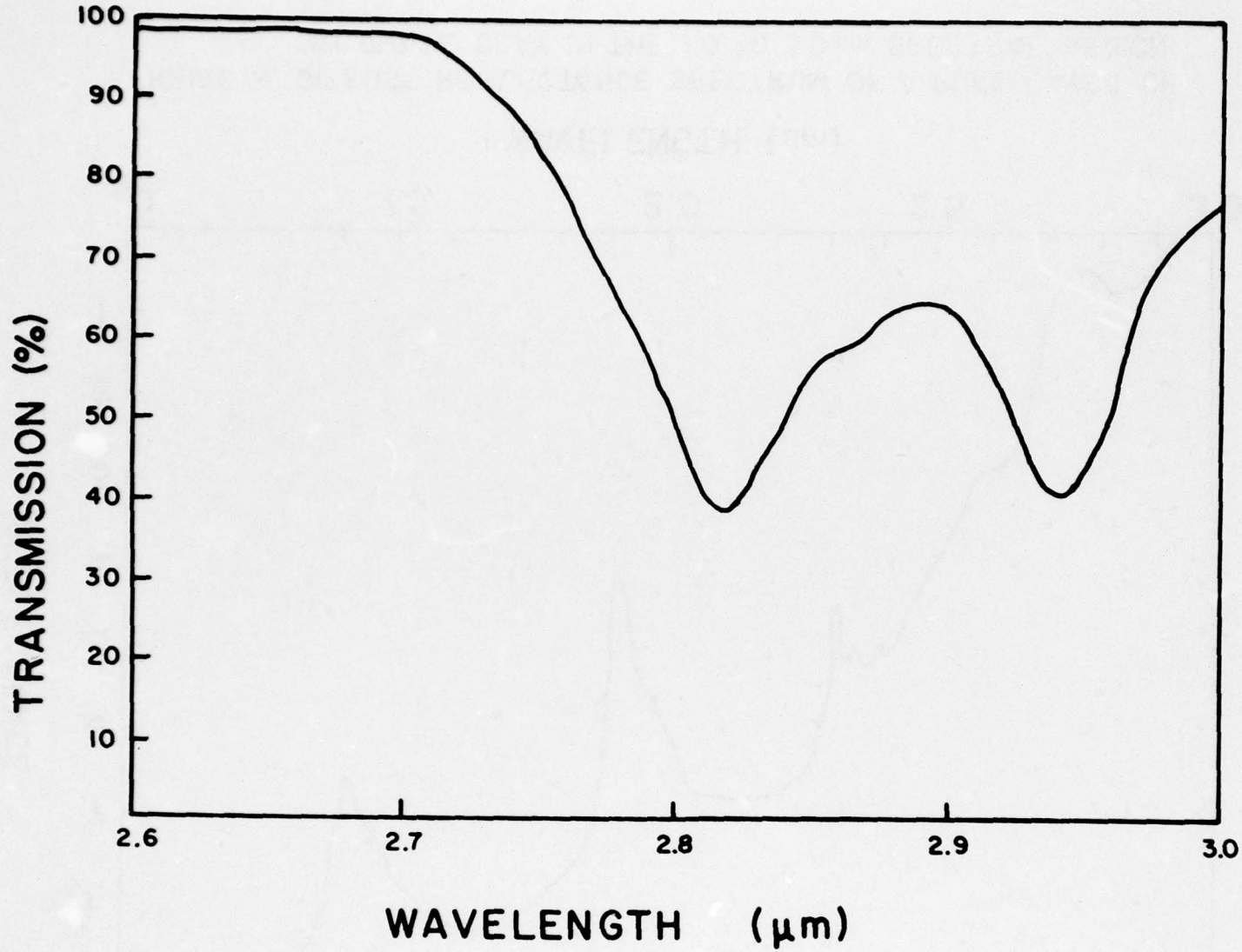

FIGURE 2. POTASSIUM BROMIDE PELLET TRANSMISSION SPECTRUM OF GROUND GYPSUM CRYSTAL IN THE 2.6 TO 3.0μm SPECTRAL REGION.

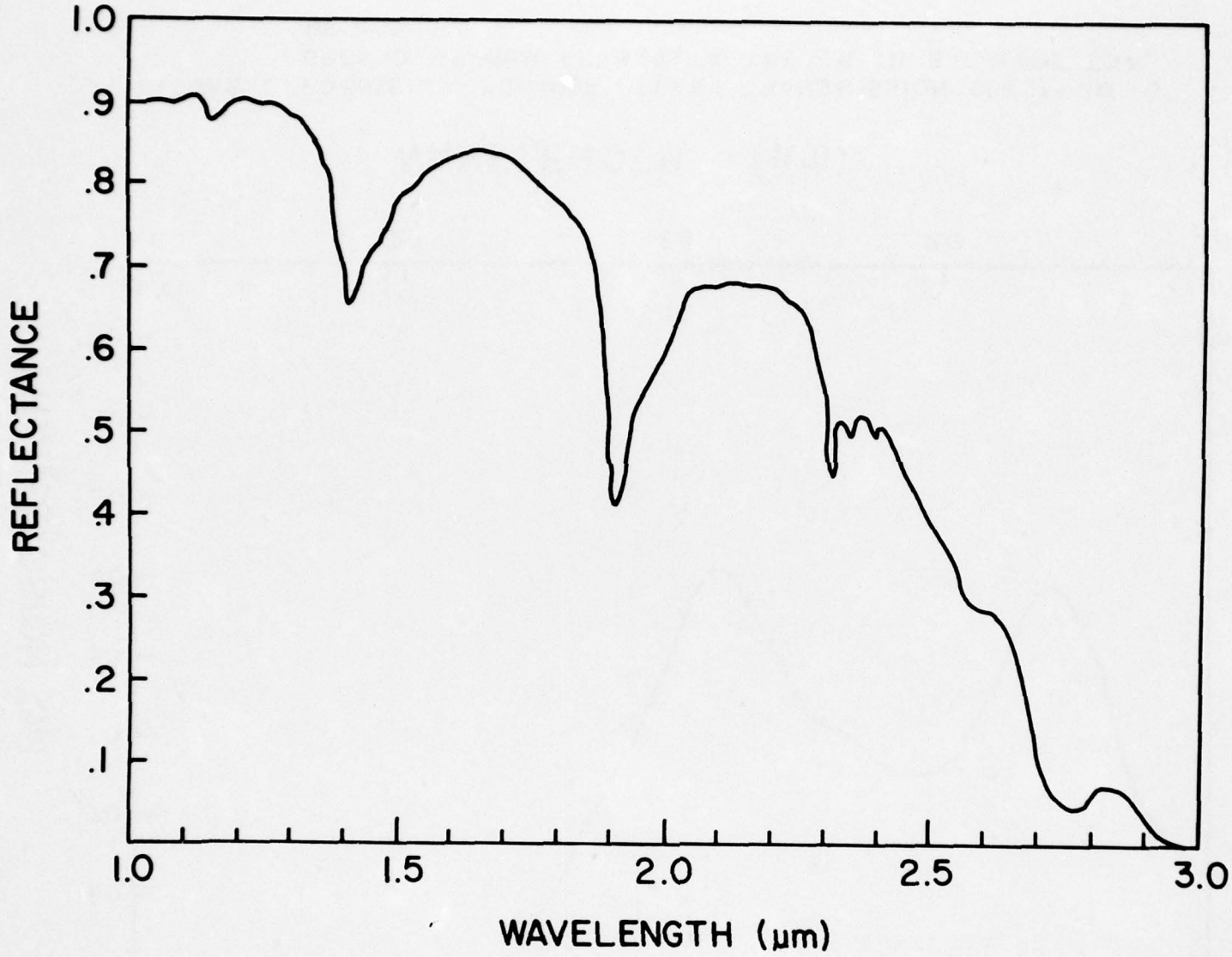

FIGURE 3. DIFFUSE REFLECTANCE SPECTRUM OF A PURE LAYER OF HECTORITE CLAY IN THE 1.0 TO 3.0 um SPECTRAL REGION.

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